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Mechanical Testing of Silicon Carbide on MISSE-7

July 15, 2012

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Space Materials Laboratory
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Prepared for:

Vice President
Technology and Laboratory Operations

Authorized by: Engineering and Technology Group

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Abstract

Silicon carbide (SiC) mechanical test specimens were included on the second Optical and Reflector Materials Experiment (ORMatE II), which was part of the seventh Materials on the International Space Station (MISSE-7) flight experiment. The SiC mechanical test specimens on the MISSE experiments were intended to determine whether LEO would degrade SiC's mechanical strength. Two suppliers provided materials that were used to prepare two kinds of flexural strength tests: modulus of rupture (a uniaxial strength test) and equibiaxial flexural strength. Samples were on orbit for approximately seventeen months, generally in an Earth-facing position. The results of strength testing of the flight samples were compared to two other sets of data for each vendor's material. Traveler samples were integrated into a flight-like tray and retained in the laboratory and were tested after the flight samples, while source material represented extra specimens that were not used to populate the flight or traveler trays and were tested before the MISSE-7 flight samples were deployed on orbit. Different statistical methods were used to compare the various datasets to determine whether the mechanical strength of the materials had been changed by LEO exposure. The conclusion was that the strength did not change as a result, which is consistent with results for SiC that had been previously flown on MISSE-6.

Acknowledgments

Dr. Iwona Palusinski was responsible for securing the participation of the SiC vendors for ORMatE-II, and was the Principal Investigator for The Aerospace Corporation on ORMatE-II. Chris Panetta assisted with post-flight sample de-integration. This work was supported in part by The Aerospace Corporation's Independent Research and Development program.

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1. Introduction

Silicon carbide (SiC) mechanical test specimens were included on the second Optical and Reflector Materials Experiment (ORMatE II), which was part of the seventh Materials on the International Space Station (MISSE-7) flight experiment. MISSE provides experimenters with low-cost access to low-Earth orbit (LEO) for studies of the effect of LEO exposure on materials. ORMatE is focused on materials for space-based optical systems. SiC has been used in such systems as an optical substrate and optical bench. Recent notable use of SiC includes the 3.5-m primary parabolic mirror of the Herschel space telescope [1]. The MISSE results are part of a larger effort to develop a space qualification for the introduction of new materials in spacecraft optical systems, especially brittle materials such as ceramics [2–4].

There are different ways to make SiC that lead to different final compositions with different material properties. In reaction-bonded SiC, a green body of SiC particles is slip cast, then infiltrated with liquid silicon to form a siliconized SiC composite. In graphite conversion SiC, a graphite form is reacted with a gaseous Si species to form SiC, which retains the porosity of the graphite or can be infiltrated with liquid silicon. Other forms of SiC that are closer in composition to single-phase include sintered or vacuum hot-pressed, which may also contain sintering aids, and vapor-deposited SiC, which is single-phase. While all of these varieties are considered SiC, their properties reflect their composition. In addition, as a brittle material, the mechanical properties of SiC will be controlled by the size and distribution of intrinsic flaws. This, in turn, will be a function of manufacturing, so even the same type of SiC may have different strengths according to processing conditions.

Previous research has shown that the mechanical [5], physical [6], and electronic [7] properties of SiC may be altered by radiation in the form of high-energy neutrons more typical of a nuclear reactor than LEO. The effect of atomic oxygen on the optical properties of polished SiC in a simulated LEO environment showed that oxidation of SiC, especially at pre-existing microcracks, degraded the reflectance of the material [8]. This latter study did not explore the possible degradation of the strength of the SiC due to oxidation of SiC to SiO_x, but if features such as these microcracks are altered by the space environment, it can be surmised that LEO exposure could degrade the strength of SiC. The relative amounts of free silicon in SiC may also contribute to changes in strength as Si is exposed to atomic oxygen in LEO.

The SiC mechanical test specimens on the MISSE experiments were intended to determine whether LEO would degrade SiC's mechanical strength. Two vendors had supplied SiC for inclusion on MISSE-6 as part of ORMatE I [9,10]; On MISSE-7, two different vendors provided SiC. Each vendor's material was used to prepare two different mechanical test specimens: Modulus of Rupture (MOR) and Equibiaxial Flexural Strength (EFS), representing uniaxial and biaxial flexural strengths of the materials, respectively. The purpose of the experiment was to compare flight samples to control specimens, and not necessarily to compare different vendors' products to each other. The data are treated as proprietary so the names of the vendors, the type of SiC they provided to MISSE-7, and the

actual strength results are not included in this report. Strength data are plotted to show comparisons between flight and traveler datasets and source material without showing actual strength values.

2. Materials and Methods

Samples placed on the ORMatE-II section of MISSE-7 were prepared as MOR rectangular beams and EFS circular disks. Exact Sample preparation and testing were conducted in accord with ASTM standards ASTM C 1161 [11] and ASTM C 1499 [12]. All specimens from Vendor 1 materials were machined at Bomas Specialty Machining, Somerville, Mass. Vendor 2 supplied biaxial disks already prepared, and MOR bars were prepared by Bomas.

The MOR bars were 50 mm long with a cross section of 3 x 4 mm, in compliance with Configuration B (B bars) of ASTM C 1161. MOR testing was conducted in a four-point bend fixture with dimensions specified by the standard. The tests were conducted at a crosshead displacement rate of 0.5 mm/min, also specified by the standard. The EFS disks prepared by Bomas from Vendor 1 material had a diameter of 31.75 mm and a thickness of 2 mm. The EFS disks supplied by Vendor 2 had a diameter of approximately 31.50 mm and a thickness of 1.5 mm. The EFS tests were conducted in a ring-on-ring fixture in which the diameters of the support and loading rings were 25.4 and 8.89 mm, respectively. The crosshead displacement rate was chosen to give an approximate loading rate of 40 MPa/s.

Samples were integrated into a flight tray and a traveler tray. Each tray contained 10 EFS disks and 15 MOR bars from each vendor. Tray construction was aluminum with a reinforced Teflon® compression layer to protect that back side of the samples. The EFS disks were separately contained in individual exposure cut-outs in the trays, while MOR bars from each vendor were arrayed side-by-side in groups in cut-outs (Figure 1). Details of the tray construction have previously been published [13]. During deployment on the space station, the sample tray containing SiC samples faced the

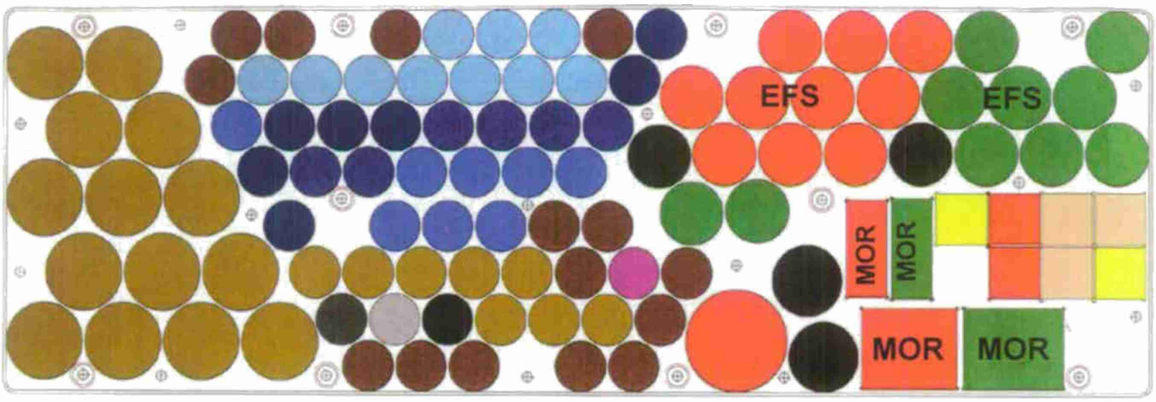

Figure 1. PEC A tray from MISSE-7. SiC specimens are shown as orange and green for the two vendors. EFS disks are in the upper right-hand quadrant of this diagram. MOR bars are contained in two separate rectangular cut-outs: the orange and green rectangles along the bottom of the tray contained 10 MOR bars from each vendor while the thinner rectangles above these and oriented along the short axis of the tray contained 5 MOR bars each.

Earth. MISSE-7 was deployed on the International Space Station during STS-129 in November 2009 and recovered during STS-134 in May 2011, for a total of approximately 17 months orbital exposure.

Samples were tested on an Instron 5500 series universal testing frame under displacement control. Fracture load was used to calculate strength usual actual sample dimensions. MOR was calculated using the following formula [11]:

$$\mathbf{S} = \frac{3\mathbf{PL}}{4\mathbf{bd}^2}, \tag{1}$$

in which P is the load at failure, L is the outer span length (40 mm), b is the specimen width (4 mm), and d is the specimen thickness (3 mm).

EFS was calculated using the following formula [12]:

$$\sigma_f = \frac{3F}{2\pi h^2}\left[(1-\nu)\frac{D_s^2 - D_L^2}{2D^2} + (1+\nu)\ln\frac{D_s}{D_L}\right], \tag{2}$$

in which F is the breaking load; h is the sample thickness; D_S, D_L, and D are the support ring, load ring, and sample diameters (25.40, 8.89 and 31.75 mm), respectively; and v is Poisson's ratio. Values for v were based on information supplied by SiC vendors or assumed based on literature data for the type of SiC being tested. For reaction-bonded SiC from Vendor 1, this value was chosen to be 0.17, while for sintered Vendor 2 material, it was 0.20.

3. Results and Analysis

A common approach to interpreting the strength of brittle materials is the two-parameter Weibull distribution [14,15]:

$$F(\sigma)=1-\exp\left[-\left(\frac{\sigma}{\sigma_\theta}\right)^m\right] \tag{3}$$

In Eq. (3), F is the probability of failure at a given stress σ, and σ_θ and m are the Weibull characteristic strength (scale factor) and Weibull modulus (shape factor), respectively. This model is based on a weakest-link theory of fracture that is consistent with the strength of a brittle material based on a random distribution of flaws placed under tensile stress. The natural logarithm of Eq. (3) can be taken twice to allow the Weibull distribution to be plotted in a linear trend according to:

$$\ln[-\ln(1-F)] = m\ln\sigma - m\ln\sigma_\theta \tag{4}$$

In a Weibull plot, the values of F associated with each test result σ_i in a sample containing n results are assumed to be regularly spaced using median rank assignment $\left[F=\frac{(i-0.5)}{n}\right]$. This is a convenient way to visualize a dataset in a Weibull plot, but it is not the appropriate way to calculate the Weibull parameters when evaluating the strength of brittle materials [16]. The Maximum Likelihood Estimate (MLE) recommended in ASTM C 1239 can be used to calculate the Weibull parameters in Eq. (3) without resorting to assuming the failure probability inherent to the median rank method, which is necessary to plot the data according to Eq. (4) or to calculate the Weibull parameters by linear regression of Eq. (4).

4. Modulus of Rupture Results

Weibull plots for MOR results from Vendors 1 and 2 are given in Figures 2 and 3, respectively, in which the plot axes are based on Eq. (4). Also plotted are the results for specimens derived from the same source material used for MISSE-7 specimens. Source material MOR datasets consist of 25 and 35 specimens for Vendors 1 and 2, respectively.

These plots show that for each vendor, the traveler and flight datasets are in good agreement, but a single Weibull distribution may not be the best way to model the strength of the data. Each vendor's

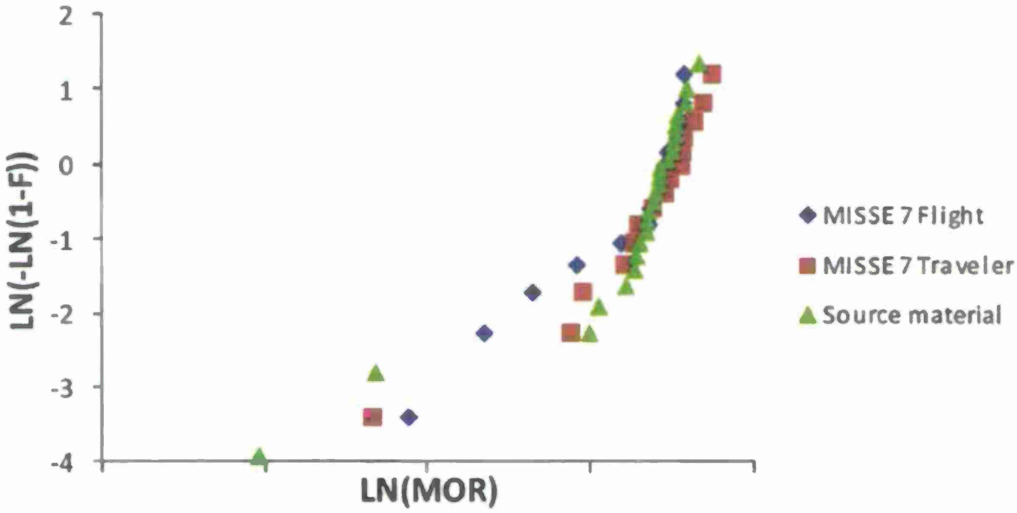

Figure 2. MOR results for Vendor 1 material.

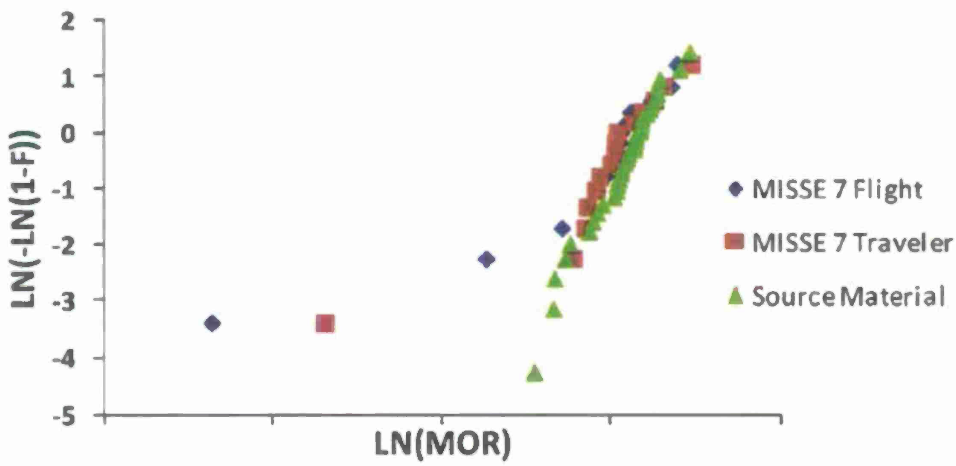

Figure 3. MOR results for Vendor 2 material.

data appear to fall into two distinct trends. Nevertheless, a non-parametric comparison of the two datasets for each vendor can be made using Anderson-Darling statistics [17,18].* Anderson-Darling tests of this type are used to determine whether pooling results from multiple material batches is statistically justified, with the implication that pooling is allowed where the different batches are drawn from the same unspecified (i.e., non-parameterized) population. In the context of the MISSE experiment, smaller sample sizes and other features of the results such as evidence for bimodal strength distributions preclude estimates of statistical models of the mechanical strength that would allow for direct comparison of statistical parameters such as mean, variance, or Weibull parameters. The Anderson-Darling tests provide a means to test the hypothesis that LEO exposure altered the flexural strength of the material on the assumption that if there were significant changes in strength one would no longer conclude that the different batches or datasets were derived from the same underlying population.

For Vendor 1 MOR data, the flight, traveler, and source material datasets all show evidence of multiple flaw populations. Use of Anderson-Darling statistics for a parameter-free comparison of all three datasets concludes that all three datasets come from the same underlying population, even if two different models might be required to completely describe the mechanical behavior of this material. The same test between only the flight samples and the source material samples also concludes that they are from the same population. In the context of evaluating the MISSE-7, this is a sufficient conclusion, but a more detailed analysis would be required to evaluate the reaction bonded SiC for specific designs or applications.

The same general approach is now taken for the MOR data for Vendor 2 SiC. Figure 3 also shows the presence of lower-strength specimens, but not necessarily a well-defined lower-strength flaw population. Use of the Anderson-Darling test for the Vendor 2 flight and traveler MOR datasets concludes that the material is from the same source population; as before, this is interpreted as indication that exposure to LEO did not change the mechanical properties.

Source material for the Vendor 2 MISSE-7 samples included an additional 35 MOR samples, which were not included in the MISSE experiment and which did not have any low-strength results. Repeating the Anderson-Darling test for three datasets (flight, traveler, and source material) led to the same result: the three samples are all from the same underlying population.

The source material depicted in Figure 3 can be well represented using a single Weibull distribution. The possibility that the low-strength results in the flight and traveler datasets could be treated as statistical outliers was investigated given the behavior of the source material. The maximum normed residual test [19] was used to identify two outliers in the flight dataset and one in the traveler dataset. These outliers are readily identified in Figure 3.

Once the outliers are removed, both Vendor 2 flight and traveler datasets can be fit using the Weibull distribution. The calculated Weibull parameters for the reduced flight and traveler datasets and the source material are shown in Table 1 where they have been normalized to the respective values of the Source Material dataset. The characteristic strengths calculated for the flight and traveler data were within one percent of the Source Material.

* Description of statistical methods and tests is given in the Appendix.

Table 1. Vendor 2 MOR Weibull Parameters

Dataset	Sample Size	Normalized m	Normalized σ_{θ}
Flight	13	0.99	1.00
Traveler	14	0.86	1.00
Source Material	35	1.00	1.00

Testing whether these Weibull distributions are statistically different was performed using the Likelihood Ratio test. This test showed no statistically significant differences when comparing the flight and traveler data or the flight, traveler, and source material in separate tests.

The conclusion to these tests is that the inclusion of the material on MISSE and exposure to the LEO environment did not affect the material properties of the sintered SiC. As seen in Figure 3, however, the outliers were found only in specimens included in MISSE, whether in flight or traveler specimens. The conclusion would thus be strengthened if the material conditions leading to the low-strength results or their cause could be determined. Limited fractography was performed on the Vendor 2 specimens to understand the nature of the fracture surface and to specifically look for anomalous conditions in the low-strength samples. The fracture surfaces showed two types of flaws related to manufacturing processes. For proprietary reasons the nature of these flaws is not discussed in this report, but one type of large flaw was found that could be considered anomalous but intrinsic to the material. Low-strength test results occurred where these larger flaws happened to be on or near the tensile surface of the specimen. Additional material and testing would be needed to generate a statistically meaningful number of low-strength tests to fully characterize the mechanical properties.

Nevertheless, these flaws are likely not due to the MISSE experiment and, in particular, not due to the LEO environment. Flaws of a similar origin, albeit of much smaller size, are likely the population responsible for the Weibull-type behavior shown in Figure 3. LEO exposure did not alter these flaws to the extent of changing the strength of the material, so similar pores of much larger size are unlikely to have been affected by LEO. Removing the outliers in Vendor 2 data to compare the datasets does not compromise the conclusion that the MOR was not affected by LEO.

5. Equibiaxial Flexural Strength

The data analysis approach for the EFS data is similar to that for the MOR data. EFS specimens from Vendor 1 were prepared from three different source disks. The number of specimens was 10 for the flight and traveler samples and 20 for the remaining source material. EFS results for all three datasets are plotted in Figure 4. These results show the same evidence of a bimodal strength population as shown in the MOR results from this vendor. The source material shows better evidence of the low-strength population since the low-strength results from flight and traveler datasets are limited to one specimen in each. These single data points are not treated as outliers because they conform to the larger trend seen in the source material and the MOR data for the vendor's material.

Vendor 1 EFS data were compared using Anderson-Darling tests as described before. Direct comparison of flight and traveler data and comparison of flight, traveler, and source material data showed that all were drawn from the same underlying population. This supports the conclusion that the LEO exposure did not significantly affect the biaxial strength of the material.

Biaxial disk specimens were received directly from Vendor 2. In addition to the ten disks integrated into each of the flight and traveler trays, an additional 32 disk specimens with similar preparation were also provided. Because these extra disks were supplied already cut and ground, they are labeled "extra disks" and not "source material" because the relationship between specimen and raw material was not verified. All three datasets are plotted together in Figure 5.

This Weibull plot shows considerable separation between the flight and traveler datasets. Statistical comparison of the flight and traveler datasets yielded different conclusions for different tests. The Anderson-Darling test for the two datasets resulted in a value of 2.37, compared to a critical value for

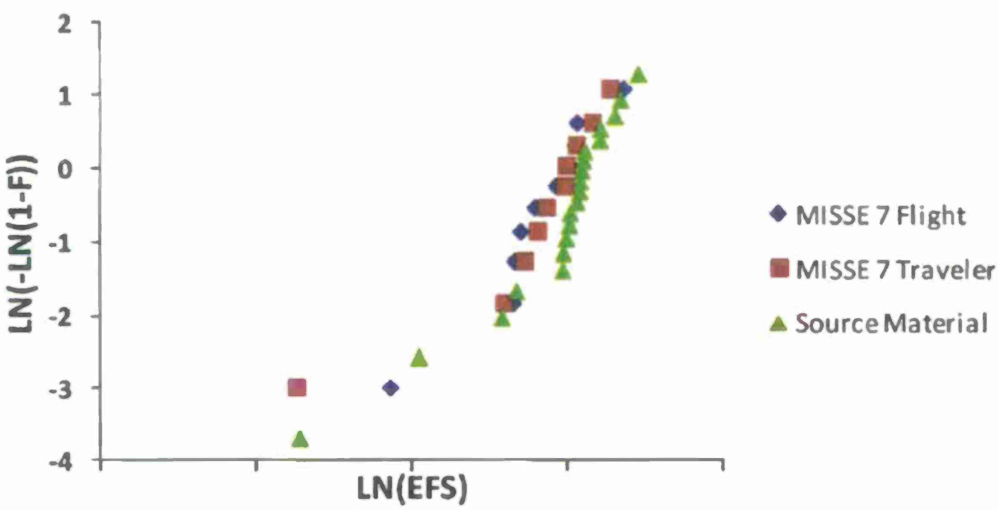

Figure 4. EFS results for Vendor 1 MISSE-7 specimens and source material.

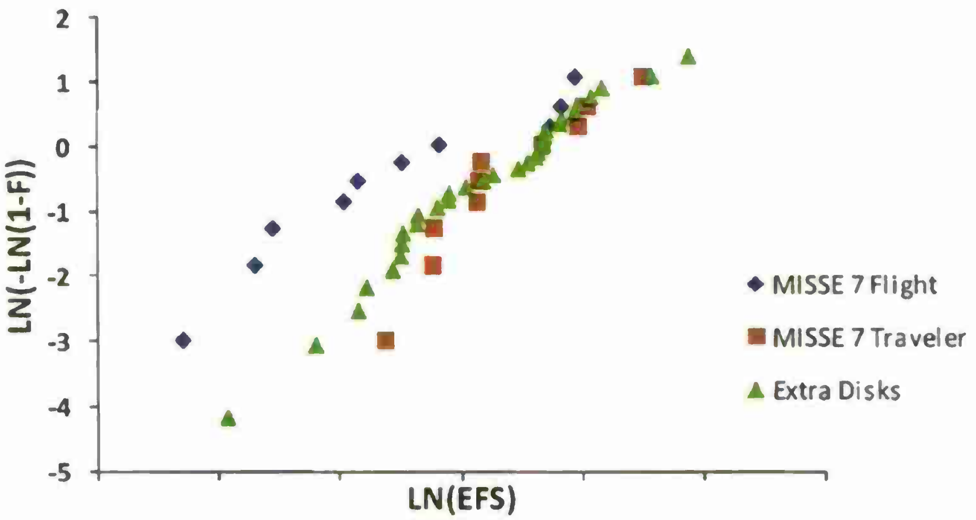

Figure 5. EFS results for Vendor 2 MISSE-7 specimens and extra disks material.

20 data points of 2.38 at the 0.05 level of significance, which indicates that the flight and traveler data come from the same underlying population (test statistic must be lower than the critical value for this conclusion). For comparison, the two-batch Anderson-Darling test of the Vendor 1 flight and traveler EFS data in Figure 5 had a test value of 0.31 compared to the same critical value. Both the flight and traveler data could be modeled using the Weibull distribution (Table 2). A likelihood ratio test returns a value of $T = 3.87$, compared to an upper-tail value of the χ^2 distribution at the 0.05 level of significance and one degree of freedom of 3.84. Thus the two Weibull distributions are statistically different at this level of significance (3.87 corresponds to a probability of 0.049). While the two tests yield different conclusions, they are both very close to critical levels of significance, adding to the uncertainty of the conclusion.

When the extra disks are added to consideration, the Anderson-Darling test for three different datasets concludes that the material is from the same underlying population. Like the flight and traveler datasets, the extra disks can be fit using a single Weibull distribution, so that a Likelihood Ratio test for the three datasets can be performed. In this case, the value of T for the three datasets is 5.78, which is less than the value of the χ^2 distribution at the 0.05 level of significance for two degrees of freedom. When the extra disks are added into consideration, the conclusions from both tests are consistent with each other and point towards no change in strength due to LEO exposure. Given a similar conclusion for the MOR data, the contrary result for the single test of Vendor 2 flight and traveler results is given less weight in asserting that LEO does not affect the strength of the Vendor 2 material.

Table 2. Vendor 2 EFS Weibull Results (normalized to Extra Disks Weibull parameters)

Dataset	Sample Size	Normalized m	σ_θ (MPa)
Flight	10	0.76	0.92
Traveler	10	1.19	0.99
Extra Disks	32	1.00	1.00

6. Conclusions

The strength of brittle materials such as SiC is typically described using the presumption that strength is controlled by the size and distribution of flaws in the material. This statistical approach typically requires a relatively large number of samples to adequately model the material using the Weibull distribution. The limit on the number of specimens imposed by a space flight experiment such as MISSE means that any model of material strength will be suspect as a means of directly measuring the effect of experimental conditions on strength.

The approach to evaluating the impact of LEO on SiC described here and previously for SiC on MISSE-6 [10] has been to compare different datasets including actual flight specimens, traveler specimens, and control or source material specimens. Where Weibull parameters can be calculated, direct comparison of datasets using the Likelihood Ratio test has been performed. In some cases, the data did not allow calculation of Weibull parameters. This usually arose where there was evidence of more than one flaw population but there were insufficient data to characterize the low-strength population. This situation required the use of a non-parametric approach using Anderson-Darling statistics. The comparison of multiple batches is used to determine whether strength test results for different batches of material can be combined to generate a statistical model for an underlying population. In the context of MISSE, this approach has been used based on the presumption that if exposure to the LEO environment had significantly altered the strength of the specimens, then a test including the flight samples would identify this change by way of a failed statistical test.

Anderson-Darling tests for the MISSE-7 data from two SiC vendors were performed in two combinations. Flight and traveler data were directly compared, and flight, traveler, and a third representative dataset (e.g., source material or extra material) were compared. In all cases, the tests indicated no significant differences. Weibull distributions could only be calculated for Vendor 2's sintered SiC. For Vendor 2 MOR MISSE data, Weibull distributions could be calculated only after removing low-strength outliers. Direct comparison of the Weibull distributions after removing outliers showed that the flight and traveler Weibull distributions were not statistically different. The nature of the flaws presumed to have caused the outlying low-strength results were not likely to have been caused by LEO exposure, so not including those results in the comparison is not likely to alter the conclusion. For Vendor 2 EFS data, there was a statistically significant difference between Weibull distributions for flight and traveler datasets, even though the Anderson-Darling test did not conclude they represented different underlying populations. The Likelihood Ratio for Vendor 2 EFS data did not reveal a significant difference when the Weibull distribution for the Vendor 2 EFS extra specimens was considered along with the flight and traveler results. The Likelihood Ratio test reflects the sample size, so the larger dataset is expected to characterize the material more accurately and dominate the test.

Three different types of SiC provided by four different vendors have been flown on MISSE-6 and -7. All results have demonstrated that LEO exposure between one and two years does not change the mechanical strength of these materials.

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Appendix—Weibull Distribution and Mathematical Methods

Weibull Distribution: The two-parameter Weibull cumulative distribution is given by:

$$P(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_\theta}\right)^m\right] \tag{I}$$

Weibull Probability Density Function: The probability density function is the derivative of the cumulative distribution (Eq. I):

$$\frac{\partial P(\sigma)}{\partial \sigma} = \frac{m}{\sigma_\theta}\left(\frac{\sigma}{\sigma_\theta}\right)^{m-1} \exp\left[-\left(\frac{\sigma}{\sigma_\theta}\right)^m\right] \tag{II}$$

Likelihood Function: For a sample of *N* data points, the likelihood function is the product of probability density function for each data point:

$$L = \prod_{i=1}^{N} \frac{m}{\sigma_\theta}\left(\frac{\sigma_i}{\sigma_\theta}\right)^{m-1} \exp\left[-\left(\frac{\sigma_i}{\sigma_\theta}\right)^m\right] \tag{III}$$

The values of m and σ_θ are estimators of the parameters from the Maximum Likelihood Method.

Log Likelihood Function: Taking the natural logarithm of Eq. III allows a sum to be substituted for the product.

Likelihood Ratio Test: One test for whether k batches are from the same population is the Likelihood Ratio Test, given by:

$$T = 2\left(L_1 + L_2 + \dots L_k - L_B\right) \le \chi^2\left(1-\alpha, k-1\right) \tag{IV}$$

In Eq. IV, L_k is the log likelihood for each batch individually and L_B is the log likelihood of all batches combined while the right-hand side of the inequality refers to the chi-squared upper tail distribution at the α level of significance with $k-1$ degrees of freedom. When comparing two batches, the number of degrees of freedom is thus 1.

Anderson-Darling Goodness of Fit Test: The Anderson-Darling test statistic is used to determine the observed significance level of the calculated Weibull model. The test statistic is given by:

$$A^2 = \left\{ \frac{-1}{n} \sum_{i=1}^{n} (2i-1)\left[\ln Z_i + \ln\left(Z_{n+1-i}\right)\right] \right\} - n, \tag{Va}$$

where n is the number of data points and

$$Z_i = \left(\frac{\sigma_i}{\sigma_\theta} \right)^m \tag{Vb}$$

The significance level (SL) of the test is given by

$$SL = \frac{1}{1 + \left[\exp\left(-0.10 + 1.24 \ln\left(A^{2*}\right) + 4.48\left(A^{2*}\right)\right)\right]} \tag{Vc}$$

Because both Weibull parameters have been estimated using MLE method (i.e., the "true" Weibull parameters are not yet known), the value of A2 is modified to A2* according to:

$$A^{2*} = \left(1 + \frac{0.2}{\sqrt{n}}\right) A^2 \tag{Vd}$$

SL is a measure of how well the data fit the Weibull model, with a maximum value of 1. If SL is less than 0.05, it is suggested that normal or lognormal distributions be used to model the data, especially if the goal is to determine a basis value [17].

Anderson-Darling Test for Combining Two Batches: The Anderson-Darling statistic (AD2) tests the hypothesis that two batches come from the same population. If the result indicates that they do, then the Weibull parameters may be estimated from the combined batches. AD2 is given by:

$$A_{mn}^2 = \frac{1}{mn} \sum_{i}^{N-1} \frac{\left(M_i N - ni\right)^2}{i\left(N-i\right)} \tag{VI}$$

In Equation VI, the two batches have m and n data points respectively, with $N = m+n$. The two datasets are combined and ranked. M_i refers to the number of data points (i.e., strength values) from the data set with m values less than or equal to the i^{th} value. For a significance level of 0.05, if AD2 is less than 2.492- 2.316/N, then the batches may be combined.

Anderson-Darling Test for Combining *k* Batches: The test statistic is given by

$$ADk = \frac{1}{N(k-1)} \sum_{i=1}^{k} (N - n_i) AD_i \tag{VIIa}$$

In Equation VIIa n_i is the size of the ith batch and N is the sum of the n_i's.

The critical value for the test is

$$CV = 1 + \frac{\left(1.25 - \frac{1.75}{\bar{n}}\right)}{\sqrt{k-1}} + \frac{0.262}{(k-1)^{0.75}}$$

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